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(54) **TREATMENT OF FABRIC ARTICLES WITH REBUILD AGENTS**

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(57) **ABSTRACT**

Methods and compositions to treat fabrics with lipophilic fluid and a rebuild agent are provided by the present invention.

**26 Claims, No Drawings**

## TREATMENT OF FABRIC ARTICLES WITH REBUILD AGENTS

### RELATED APPLICATIONS

This application claims priority to U.S. Provisional Application Serial No. 60/342,714 filed on Dec. 20, 2001.

### FIELD OF THE INVENTION

The present invention relates to compositions and methods to treat fabrics with a lipophilic fluid and a rebuild agent.

### BACKGROUND OF THE INVENTION

Conventional laundering techniques for the cleaning and treatment of fabric articles such as garments have long involved both traditional aqueous based washing and a technique commonly referred to as "dry cleaning". Traditional aqueous based washing techniques have involved immersion of the fabric articles in a solution of water and detergent or soap products followed by rinsing and drying. However, such conventional immersion cleaning techniques have proven unsatisfactory on a wide range fabric articles that require special handling and/or cleaning methods due to fabric content, construction, etceteras, that is unsuitable for immersion in water.

Accordingly, the use of the laundering method of "dry cleaning" has been developed. Dry cleaning typically involves the use of non-aqueous, lipophilic fluids as the solvent or solution for cleaning. While the absence of water permits the cleaning of fabrics without the potential disastrous side effects water may present, these lipophilic fluids do not perform well on hydrophilic and/or combination soils.

Because these lipophilic fluids are typically used in "neat" form (i.e. they contain no additional additives), dry cleaners must often perform pre-treating and/or pre-spotting to remove tough soils from fabrics prior to the dry cleaning cycle. Further, nothing is typically added to boost "whiteness" or "brightness" in fabrics that are dry-cleaned as can be observed from "dingy" or "dull" fabrics returned from a dry cleaner. It would be desirable to add bleaching to the lipophilic fluid treatment regimen in order to increase the lipophilic fluids' brightening, whitening, and/or soil removal capability thereby reducing or eliminating the need for pre-treating and/or pre-spotting.

Many fabrics and textiles highly valued by the consumer (e.g., silk) are prone to undue damage when exposed to water in large quantities. For this reason garments made from such fabric and textiles must be dry cleaned.

Accordingly, the need remains for fabric care and/or treatment regimens for use with lipophilic fluid compositions that incorporate fabric care actives.

### SUMMARY OF THE INVENTION

This need is met by the present invention wherein fabric care active-containing care and treatment regimens and compositions for use with lipophilic fluid compositions are provided.

The present invention is directed to a method for attaining improved fabric cleaning in a lipophilic fluid treatment regimen, wherein the method includes the steps of exposing the fabric to a lipophilic fluid and exposing the fabric to a rebuild agent.

The present invention is also directed to a composition for attaining improved fabric cleaning in a lipophilic fluid

treatment regimen, wherein the composition includes a lipophilic fluid and a rebuild agent.

These and other aspects, features and advantages will become apparent to those of ordinary skill in the art from a reading of the following detailed description and the appended claims. All percentages, ratios and proportions herein are by weight, unless otherwise specified. All temperatures are in degrees Celsius ( $^{\circ}$  C.) unless otherwise specified. All measurements are in SI units unless otherwise specified. All documents cited are in relevant part, incorporated herein by reference.

### DETAILED DESCRIPTION OF THE INVENTION

#### Definitions

The term "fabrics" and "fabric" used herein is intended to mean any article that is customarily cleaned in a conventional laundry process or in a dry cleaning process. As such the term encompasses articles of clothing, linen, drapery, and clothing accessories. The term also encompasses other items made in whole or in part of fabric, such as tote bags, furniture covers, tarpaulins and the like.

The term "soil" means any undesirable substance on a fabric article that is desired to be removed. By the terms "water-based" or "hydrophilic" soils, it is meant that the soil comprised water at the time it first came in contact with the fabric article, or the soil retains a significant portion of water on the fabric article. Examples of water-based soils include, but are not limited to beverages, many food soils, water soluble dyes, bodily fluids such as sweat, urine or blood, outdoor soils such as grass stains and mud.

#### Lipophilic Fluid

The lipophilic fluid herein is one having a liquid phase present under operating conditions of a fabric article treating appliance, in other words, during treatment of a fabric article in accordance with the present invention. In general such a lipophilic fluid can be fully liquid at ambient temperature and pressure, can be an easily melted solid, e.g., one which becomes liquid at temperatures in the range from about 0 deg. C. to about 60 deg. C., or can comprise a mixture of liquid and vapor phases at ambient temperatures and pressures, e.g., at 25 deg. C. and 1 atm. pressure. Thus, the lipophilic fluid is not a compressible gas such as carbon dioxide.

It is preferred that the lipophilic fluids herein be nonflammable or have relatively high flash points and/or low VOC (volatile organic compound) characteristics, these terms having their conventional meanings as used in the dry cleaning industry, to equal or, preferably, exceed the characteristics of known conventional dry cleaning fluids.

Moreover, suitable lipophilic fluids herein are readily flowable and nonviscous.

In general, lipophilic fluids herein are required to be fluids capable of at least partially dissolving sebum or body soil as defined in the test hereinafter. Mixtures of lipophilic fluid are also suitable, and provided that the requirements of the Lipophilic Fluid Test, as described below, are met, the lipophilic fluid can include any fraction of dry-cleaning solvents, especially newer types including fluorinated solvents, or perfluorinated amines. Some perfluorinated amines such as perfluorotributylamines while unsuitable for use as lipophilic fluid may be present as one of many possible adjuncts present in the lipophilic fluid-containing composition.

Other suitable lipophilic fluids include, but are not limited to, diol solvent systems e.g., higher diols such as C6- or C8- or higher diols, organosilicone solvents including both cyclic and acyclic types, and the like, and mixtures thereof.

A preferred group of nonaqueous lipophilic fluids suitable for incorporation as a major component of the compositions of the present invention include low-volatility nonfluorinated organics, silicones, especially those other than amino functional silicones, and mixtures thereof. Low volatility nonfluorinated organics include for example OLEAN® and other polyol esters, or certain relatively nonvolatile biodegradable mid-chain branched petroleum fractions.

Another preferred group of nonaqueous lipophilic fluids suitable for incorporation as a major component of the compositions of the present invention include, but are not limited to, glycol ethers, for example propylene glycol methyl ether, propylene glycol n-propyl ether, propylene glycol t-butyl ether, propylene glycol n-butyl ether, dipropylene glycol methyl ether, dipropylene glycol n-propyl ether, dipropylene glycol t-butyl ether, dipropylene glycol n-butyl ether, tripropylene glycol methyl ether, tripropylene glycol n-propyl ether, tripropylene glycol t-butyl ether, tripropylene glycol n-butyl ether. Suitable silicones for use as a major component, e.g., more than 50%, of the composition include cyclopentasiloxanes, sometimes termed "D5", and/or linear analogs having approximately similar volatility, optionally complemented by other compatible silicones. Suitable silicones are well known in the literature, see, for example, Kirk Othmer's Encyclopedia of Chemical Technology, and are available from a number of commercial sources, including General Electric, Toshiba Silicone, Bayer, and Dow Corning. Other suitable lipophilic fluids are commercially available from Procter & Gamble or from Dow Chemical and other suppliers.

#### Qualification of Lipophilic Fluid and Lipophilic Fluid Test (LF Test)

Any nonaqueous fluid that is both capable of meeting known requirements for a dry-cleaning fluid (e.g, flash point etc.) and is capable of at least partially dissolving sebum, as indicated by the test method described below, is suitable as a lipophilic fluid herein. As a general guideline, perfluorobutylamine (Fluorinert FC-43®) on its own (with or without adjuncts) is a reference material which by definition is unsuitable as a lipophilic fluid for use herein (it is essentially a nonsolvent) while cyclopentasiloxanes have suitable sebum-dissolving properties and dissolves sebum.

The following is the method for investigating and qualifying other materials, e.g., other low-viscosity, free-flowing silicones, for use as the lipophilic fluid. The method uses commercially available Crisco® canola oil, oleic acid (95% pure, available from Sigma Aldrich Co.) and squalene (99% pure, available from J. T. Baker) as model soils for sebum. The test materials should be substantially anhydrous and free from any added adjuncts, or other materials during evaluation.

Prepare three vials, each vial will contain one type of lipophilic soil. Place 1.0 g of canola oil in the first; in a second vial place 1.0 g of the oleic acid (95%), and in a third and final vial place 1.0 g of the squalene (99.9%). To each vial add 1 g of the fluid to be tested for lipophilicity. Separately mix at room temperature and pressure each vial containing the lipophilic soil and the fluid to be tested for 20 seconds on a standard vortex mixer at maximum setting. Place vials on the bench and allow to settle for 15 minutes at room temperature and pressure. If, upon standing, a clear single phase is formed in any of the vials containing lipophilic soils, then the nonaqueous fluid qualifies as suitable for use as a "lipophilic fluid" in accordance with the present invention. However, if two or more separate layers are formed in all three vials, then the amount of nonaqueous fluid dissolved in the oil phase will need to be further

determined before rejecting or accepting the nonaqueous fluid as qualified.

In such a case, with a syringe, carefully extract a 200-microliter sample from each layer in each vial. The syringe-extracted layer samples are placed in GC auto sampler vials and subjected to conventional GC analysis after determining the retention time of calibration samples of each of the three models soils and the fluid being tested. If more than 1% of the test fluid by GC, preferably greater, is found to be present in any one of the layers which consists of the oleic acid, canola oil or squalene layer, then the test fluid is also qualified for use as a lipophilic fluid. If needed, the method can be further calibrated using heptacosafuorotributylamine, i.e., Fluorinert FC-43 (fail) and cyclopentasiloxane (pass). A suitable GC is a Hewlett Packard Gas Chromatograph HP5890 Series II equipped with a split/splitless injector and FID. A suitable column used in determining the amount of lipophilic fluid present is a J&W Scientific capillary column DB-1HT, 30 meter, 0.25 mm id, 0.1 um film thickness cat#1221131. The GC is suitably operated under the following conditions:

Carrier Gas: Hydrogen

Column Head Pressure: 9 psi

Flows: Column Flow @~1.5 ml/min.

Split Vent @~250-500 ml/min.

Septum Purge @ 1 ml/min.

Injection: HP 7673 Autosampler, 10 ul syringe, 1 ul injection

Injector Temperature: 350° C.

Detector Temperature: 380° C.

Oven Temperature Program: initial 60° C. hold 1 min. rate 25° C./min.

final 380° C. hold 30 min.

Preferred lipophilic fluids suitable for use herein can further be qualified for use on the basis of having an excellent garment care profile. Garment care profile testing is well known in the art and involves testing a fluid to be qualified using a wide range of garment or fabric article components, including fabrics, threads and elastics used in seams, etc., and a range of buttons. Preferred lipophilic fluids for use herein have an excellent garment care profile, for example they have a good shrinkage and/or fabric puckering profile and do not appreciably damage plastic buttons. Certain materials which in sebum removal qualify for use as lipophilic fluids, for example ethyl lactate, can be quite objectionable in their tendency to dissolve buttons, and if such a material is to be used in the compositions of the present invention, it will be formulated with water and/or other solvents such that the overall mix is not substantially damaging to buttons. Other lipophilic fluids, D5, for example, meet the garment care requirements quite admirably. Some suitable lipophilic fluids may be found in granted U.S. Pat. Nos. 5,865,852; 5,942,007; 6,042,617; 6,042,618; 6,056,789; 6,059,845; and 6,063,135, which are incorporated herein by reference.

Lipophilic fluids can include linear and cyclic polysiloxanes, hydrocarbons and chlorinated hydrocarbons, with the exception of PERC and DF2000 which are explicitly not covered by the lipophilic fluid definition as used herein. More preferred are the linear and cyclic polysiloxanes and hydrocarbons of the glycol ether, acetate ester, lactate ester families. Preferred lipophilic fluids include cyclic siloxanes having a boiling point at 760 mm Hg. of below about 250° C. Specifically preferred cyclic siloxanes for use in this invention are octamethylcyclotetrasiloxane,

decamethylcyclopentasiloxane, and dodecamethylcyclohexasiloxane. Preferably, the cyclic siloxane comprises decamethylcyclopentasiloxane (D5, pentamer) and is substantially free of octamethylcyclotetrasiloxane (tetramer) and dodecamethylcyclohexasiloxane (hexamer).

However, it should be understood that useful cyclic siloxane mixtures might contain, in addition to the preferred cyclic siloxanes, minor amounts of other cyclic siloxanes including octamethylcyclotetrasiloxane and hexamethylcyclotrisiloxane or higher cyclics such as tetradecamethylcycloheptasiloxane. Generally the amount of these other cyclic siloxanes in useful cyclic siloxane mixtures will be less than about 10 percent based on the total weight of the mixture. The industry standard for cyclic siloxane mixtures is that such mixtures comprise less than about 1% by weight of the mixture of octamethylcyclotetrasiloxane.

#### Rebuild Agents

Specific nonlimiting examples of suitable rebuild agents for use in the present invention are described in WO 01/72944 (US 20010036907A1), WO 01/72940, WO 01/72939.

In one embodiment, the rebuild agent used in the present invention is water-soluble or water-dispersible in nature and in a preferred form comprises a polymeric backbone having one or more pendant groups which undergo the chemical change to cause an increase in affinity for fabric.

The weight average molecular weight ( $M_w$ ) of the rebuild agent (as determined by GPC) may typically be in the range of from about 500 to about 2,000,000 for example 1,000 to 1,500,000. Preferably though, it is from 1,000 to 100,000, more preferably from 5,000 to 50,000, especially from 10,000 to 15,000.

By water-soluble, as used herein, what is meant is that the material forms an isotropic solution on addition to water or another aqueous solution.

By water-dispersible, as used herein, what is meant is that the material forms a finely divided suspension on addition to water or another aqueous solution. Preferably though, the term "water-dispersible" means that the material, in water at pH 7 and at 250C., produces a solution or a dispersion having long-term stability.

By an increase in the affinity of the material for the fabric upon a chemical change, what is meant is that at sometime during the laundry treatment process, the amount of material that has been deposited is greater when the chemical change is occurring or has occurred, compared to when the chemical change has not occurred and is not occurring, or is occurring more slowly, the comparison being made with all conditions being equal except for that change in the conditions which is necessary to affect the rate of chemical change.

Deposition includes adsorption, cocrystallisation, entrapment and/or adhesion.

In a first aspect of the invention, the polymeric backbone of the rebuild agent may be of a similar chemical structure to that of at least some of the fibers of the fabric onto which it is to be deposited. For example, if the fabric is cellulosic in nature, e.g. cotton, the polymeric backbone preferably comprises cellulose and/or a cellulose derivative or another 1,4-linked polysaccharide having an affinity for cellulose, such as mannan and/or glucomannan.

In a second aspect of the invention, the polysaccharide may include substitutions. The average degree of substitution on the polysaccharide of any pendant groups which are capable of undergoing a chemical change (plus any non-functional pendant groups which may be present) is preferably from 0.3 to 3, more preferably from 0.4 to 1. Still more preferred is a degree of substitution of from 0.5 to 0.75.

The polysaccharide may be straight or branched. Many naturally occurring polysaccharides have at least some degree of branching, or at any rate, at least some saccharide rings are in the form of pendant side groups (and therefore are not in themselves counted in the degree of substitution) on a main polysaccharide backbone.

A polysaccharide comprises a plurality of saccharide rings, which have pendant hydroxyl groups. The pendant groups can be bonded chemically or by other bonding mechanism, to these hydroxyl groups by any means described herein below. The "average degree of substitution" means the average number of pendant groups per saccharide ring for the totality of polysaccharide molecules in the sample and is determined for all saccharide rings whether they form part of a linear backbone or are themselves, pendant side groups in the polysaccharide. Other polymeric backbones suitable for polymeric material for use in the present invention include those described in Hydrocolloid Applications, A. Nussinswitch, Blackie 1997.

Pendant groups which are capable of undergoing a chemical change, which causes the increased fabric affinity as exhibited by the pendant group will usually undergo hydrolysis and/or perhydrolysis and/or bond-cleavage, which may be catalyzed by an enzyme or another catalyst.

Hydrolysis of ester-linked groups is most typical. However, preferably this change is not merely protonation or deprotonation, i.e. a pH induced effect. The chemical change may occur in or to a group covalently bonded to a polymeric backbone, especially, the loss of one or more such groups. These group(s) is/are pendant on the backbone.

In the case of the first aspect of the invention these are ester-linked groups based on monocarboxylic acids.

The second aspect of the invention is not limited to (but may include) use of rebuild agents incorporating ester linkages based on monocarboxylic acids. Mono-, di- and polycarboxylic ester- or semi-ester-linkages, ester and semi-ester linkages derived from non-carboxylic acids, as well as carbamate, urea or silyl linked groups, as well as others, are also possible. Particularly preferred are cellulose monoacetate, cellulose hemisuccinate, and cellulose 2-(2-hydroxy-1-oxopropoxy)propanoate. The term cellulose monoacetate is used herein to denote those acetates with the degree of substitution of 1 or less.

The degrees of substitution for the totality of all pendant substituents in the following order of increasing preference: from 0.3 to 3, from 0.4 to 1, from 0.5 to 0.75, from 0.6 to 0 However, as well as the groups which undergo the chemical change, pendant groups of other types may optionally be present, i.e. groups which do not undergo a chemical change to enhance fabric affinity. Within that class of other groups is the sub-class of groups for enhancing the solubility of the rebuild agent (e.g. groups which are, or contain one or more free carboxylic acid/salt and/or sulfonic acid/salt and/or sulfate groups). Examples of solubility enhancing substituents include carboxyl, sulfonyl, hydroxyl, (poly) ethyleneoxy-and/or(poly)propyleneoxy-containing groups, as well as amine groups.

The other pendant groups may constitute from 0% to 65%, more preferably from 0% to 10% (e.g. from 0% to 5%) of the total number of pendant groups. The minimum number of other pendant groups may, for example be 0.1% or 1% of the total. The water-solubilizing groups could comprise from 0% to 100% of those other groups but preferably from 0% to 20%, more preferably from 0% to 10%, still more preferably from 0% to 5% of the total number of other pendant groups.

Those rebuild agents used in the present invention which are not commercially available may be prepared by a

number of different synthetic routes, for example:-(1) polymerization of suitable monomers, for example, enzymatic polymerization of saccharides, e.g. per S. Shoda, & S. Kobayashi, *Makromol. Symp.* 1995, 99, 179–184 or oligosaccharide synthesis by orthogonal glycosylation e.g. per H. Paulsen, *Angew. Chem. Int. Ed. Engl.* 1995, 34, 1432–1434.;-17-(2) derivatization of a polymeric backbone (either naturally occurring, especially polysaccharides, especially beta-1,4-linked polysaccharides, especially cellulose, mannan, glucomannan, galactomannan, xyloglucan; or synthetic polymers) up to the required degree of substitution with functional groups which improve the solubility of the polymer using a reagent (especially acid halides, especially carboxylic acid halides, anhydrides, carboxylic acid anhydrides, carboxylic acids or, carbonates) in a solvent which either dissolves the backbone, swells the backbone, or does not swell the backbone but dissolves or swells the product; (3) hydrolysis of polymer derivatives (especially esters) down to the required degree of substitution; or (4) a combination of any two or more of routes (1)–(3).

The degree and pattern of substitution from routes (1) or (2) may be subsequently altered by partial removal of functional groups by hydrolysis or solvolysis or other cleavage. Relative amounts of reactants and reaction time scan also be used to control the degree of substitution. In addition, or alternatively, the degree of polymerization of the backbone may be reduced before, during, or after the derivatization with functional groups. The degree of polymerization of the backbone may be increased by further polymerization or by cross linking agents before, during, or after the derivatization step.

Cellulose esters of hydroxyacids can be obtained using the acid anhydride, typically in acetic acid solution. When the product has dissolved the liquid is poured into water. Glycollic and lactic esters can be made in this way. Cellulose glycollate may also be obtained from cellulosechloracetate (B.P. 320,842) by treating 100 parts with 32 parts of NaOH in alcohol added in small portions.

An alternative method of preparing cellulose esters consists in the partial displacement of the acid radical in a cellulose ester by treatment with another acid of higher ionization constant. The ester is heated at about 1000 with the acid which, preferably, should be a solvent for the ester. By this means cellulose acetate-oxalate, tartrate, maleate, pyruvate, salicylate and phenylglycollate have been obtained, and from cellulose tribenzoate a cellulose benzoate-pyruvate. A cellulose acetate-lactate or acetate-glycollate could be made in this way also. As an example cellulose acetate (10 g) in dioxan (75 ml) containing oxalic acid (10 g) is heated at 1000 for 2 hours under reflux.

Multiple esters are prepared by variations of this process. A simple ester of cellulose, e.g. the acetate, is dissolved in a mixture of two (or three) organic acids, each of which has an ionization constant greater than that of acetic acid ( $1.82 \times 10^{-5}$ ). With solid acids suitable solvents such as propionic acid, dioxan and ethylene dichloride are used. If a mixed cellulose ester is treated with an acid this should—19 have an ionization constant greater than that of either of the acids already in combination.

A cellulose acetate-lactate-pyruvate is prepared from cellulose acetate, 40 per cent. acetyl (100 g), in a bath of 125 ml pyruvic acid and 125 ml of 85 per cent lactic acid by heating at 1000 for 18 hours. The product is soluble in water and is precipitated and washed with ether-acetone.

#### SYNTHETIC EXAMPLES FOR MAKING REBUILD AGENTS

##### EXAMPLE 1

Preparation of Cellulose “Monoacetate” This was prepared by the methods of WO 91/16359.

##### EXAMPLE 1a

30.0 g of cellulose diacetate (DS 2.45) (the starting cellulose ester), 0.08 g of molybdenum carbonyl (catalyst), 213.6 g of methanol (reactive solvent 1) and 30.0 g of water (reactive solvent 2) are loaded into a 1-liter, steel Parr reactor equipped with a magnetically coupled agitator. The reactor is sealed, then heated to 140° C. The heat-up time is typically 1 to 2 hours. The initial pressure in the reactor is typically 200 500 psi (1379 3447 kPa) nitrogen. The reaction mixture is stirred at 140° C. for 7 hours. Then the reaction mixture is allowed to cool to room temperature, which typically takes 2 to 3 hours. The products are isolated by filtration of the resulting slurry. The reactive solvent, as well as by-products such as methyl acetate, can be recovered from the filtrate by distillation. The product is cellulose monoacetate and the yield is 66%. The key analyses are: DS=0.48; intrinsic viscosity (0.25 g per 100 ml of DMSO)=0.55.

##### EXAMPLE 1b

30.0 g of cellulose diacetate (DS 2.45) (the starting cellulose ester), 0.05 g of molybdenum (VI) oxide and 237.3 g of methanol (reactive solvent) are loaded into a 1-liter, steel Parr reactor equipped with a magnetically coupled agitator. The reactor is sealed, then heated to 1550 C. The heat-up time is typically 1 to 2 hours. The initial pressure in the reactor is typically 200 500 psi (1379 3447 kPa) nitrogen. The reaction mixture is stirred at 155° C. for 3 hours. Then the reaction mixture is allowed to cool to room temperature, which typically takes 2 to 3 hours. The products are isolated by filtration of the resulting slurry. The reactive solvent, as well as certain by-products such as methyl acetate can be recovered from the filtrate by distillation. The product is cellulose monoacetate and the yield is 87%. The key analyses are: DS=0.50; intrinsic viscosity (0.25 g per 100 ml of DMSO)=1.16.

##### EXAMPLE 2

###### Preparation of Cellulose Hemisuccinate (First Route)

Cellulose hemisuccinate was prepared following B.P. 410,125. A mixture of cellulose (Whatman cellulose powder CF11 which is cotton, 5 g), succinic anhydride (25 g), and pyridine (75 ml) was kept at 65° C. for a week. On pouring into methanol the pyridinium salt of cellulose hemisuccinate was obtained. The crude cellulose hemisuccinate, pyridinium salt, was washed repeatedly with methanol to remove pyridine and unused reactants. The pyridinium salt of cellulose hemisuccinate was converted to the free acid form by driving off the pyridine under vacuum at <95° C.

Infrared spectra of reagents and products were recorded on a Bio-Rad FTS-7 infrared spectrometer using a Graseby Specac (Part #10500) Single Reflection Diamond ATR attachment. The degree of substitution of cellulose hemisuccinate prepared from cotton fibres was determined by a one-step neutralisation of the carboxylic acid groups and hydrolysis of the ester groups, using an excess of sodium hydroxide, followed by titration of the excess sodium hydroxide with a standard solution of hydrochloric acid, using phenolphthalein as an indicator. The figure thus obtained was 2.8.

The infrared spectrum of the product in its neutralised, sodium salt form, has two distinct bands attributable to the stretching of C—O. The band at 1574 cm<sup>-1</sup> is attributable to carboxylate anion, a band for which is expected at 1550–1610 cm<sup>-1</sup>. It is therefore reasonable to attribute the

other band at 1727 cm<sup>-1</sup> to ester, a band for which is expected at 1735–1750 cm<sup>-1</sup>. The infrared spectrum is therefore consistent with a hemiester salt.

#### EXAMPLE 3

##### Preparation of Cellulose Hemisuccinate (Route 2)

Cellulose hemisuccinate was prepared following GB-A-410,125. A mixture of cellulose (Avicel PH105, 5 g), succinic anhydride (25 g), and pyridine (75 ml) was kept at 65° C. for a week. On pouring into methanol the pyridinium salt of cellulose hemisuccinate was obtained. The crude cellulose hemisuccinate, pyridinium salt, was washed repeatedly with methanol to remove pyridine and unused reactants.

When this gel was mixed with dilute aqueous sodium hydroxide, it did not immediately dissolve but remained as lumps, but it did slowly dissolve to form a near-optically-clear solution. The fact that the methanol-washed cellulose hemisuccinate was not immediately soluble in dilute aqueous sodium hydroxide indicated that the cellulose hemisuccinate was slightly cross linked. The methanol-rinsed cellulose hemisuccinate was used to prepare a cellulose hemisuccinate having a lower degree of substitution and with fewer cross links which was water dispersable. A homogeneous solution was prepared by partially hydrolysing the cellulose hemisuccinate as follows. Cellulose hemisuccinate prepared from microcrystalline cellulose, in the form of a gel of cellulose hemisuccinate, pyridinium salt, dispersed in methanol, was added to 50 ml of stirred 0.1 M NaCl solution at 50° C. 0.1 M NaOH solution was added until the pH was raised to 7.0 (18.0 ml was required). More 0.1 M NaOH solution was added until the pH was raised to ~10.5 (3.0 ml was required). This pH was then maintained for 45 minutes by further additions of 0.1 M NaOH solution (4.2 ml was required). The mixture was then cooled to room temperature and neutralised using 1.0 M HCl (0.18 ml was required). After this procedure the solution was only slightly turbid. The polymer was separated from inorganic salts by ultrafiltration (Amicon, Inc.) employing a cellulose triacetate membrane with a molecular weight cut-off of 10,000 (Sartorius SM 145 39).

The degree of substitution of cellulose hemisuccinate prepared from by this route was determined by a one-step neutralisation of the carboxylic acid groups and hydrolysis of the ester groups, using an excess of sodium hydroxide, followed by titration of the excess sodium hydroxide with a standard solution of hydrochloric acid, using phenolphthalein as an indicator. The figure thus obtained was 2.0.

#### EXAMPLE 4

##### Preparation of Cellulose 2-(2-hydroxy-1-oxopropoxy)propanoate

Following the method described in DE 3,322,118 a mixture of 2.33 g lactide (3,6-dimethyl-1,4-dioxane -2,5-dione) and 29.7 g of cellulose solution (obtained by dissolving 14 g of microcrystalline cellulose (Avicel PH105) swollen with 14 g of N,N-dimethylacetamide in a mixture of 200 ml of N,N-dimethylacetamide and 16.8 g of lithium chloride) was treated with 1.5 ml of triethyl amine and stirred at 75° C. for 1.5 hours.

Cellulose 2-(2-hydroxy-1-oxopropoxy)propanoate was isolated by pipetting the reaction mixture into 300 ml of methanol. The product gel was washed with a further two batches of 300 ml of methanol. At this stage the methanol-swollen 2-(2-hydroxy-1-oxopropoxy)propanoate was water soluble.

The cellulose 2-(2-hydroxy-1-oxopropoxy)propanoate was dried in a vacuum oven at room temperature. The dry cellulose 2-(2-hydroxy-1-oxopropoxy)propanoate was partially soluble.

In another embodiment, the average molecular weight of the rebuild agent (as determined by GPC) is in the range 12,000 to 20,000, preferably 15,000 to 20,000.

The rebuild agents may be incorporated into compositions containing only a diluent and/or also comprising another active ingredient. The compound is typically included in said compositions at levels of from 0.005% to 25% by weight; preferably 0.01% to 10%; most preferably 0.025% to 2.5%.

##### Adjunct Ingredients

In addition to the rebuild agents described above, other fabric care actives such as adjunct materials may be used in the methods and compositions of the present invention.

Adjunct materials can vary widely and can be used at widely ranging levels. For example, detergent enzymes such as proteases, amylases, cellulases, lipases and the like as well as bleach catalysts including the macrocyclic types having manganese or similar transition metals all useful in laundry and cleaning products can be used herein at very low, or less commonly, higher levels. Adjunct materials that are catalytic, for example enzymes, can be used in “forward” or “reverse” modes, a discovery independently useful from the specific appliances of the present invention. For example, a lipolase or other hydrolase may be used, optionally in the presence of alcohols as adjuncts, to convert fatty acids to esters, thereby increasing their solubility in the lipophilic fluid. This is a “reverse” operation, in contrast with the normal use of this hydrolase in water to convert a less water-soluble fatty ester to a more water-soluble material. In any event, any adjunct ingredient must be suitable for use in combination with the lipophilic fluid.

The compositions may comprise emulsifiers. Emulsifiers are well known in the chemical art. Essentially, an emulsifier acts to bring two or more insoluble or semi-soluble phases together to create a stable or semi-stable emulsion. It is preferred in the claimed invention that the emulsifier serves a dual purpose wherein it is capable of acting not only as an emulsifier but also as a treatment performance booster. For example, the emulsifier may also act as a surfactant thereby boosting cleaning performance. Both ordinary emulsifiers and emulsifier/surfactants are commercially available.

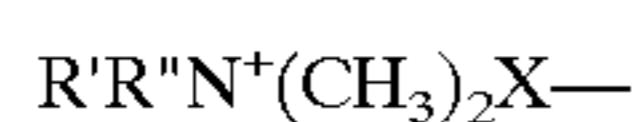
Some suitable cleaning additives (adjunct ingredients) include, but are not limited to, builders, surfactants, enzymes, bleach activators, bleach catalysts, bleach boosters, bleaches, alkalinity sources, antibacterial agents, colorants, perfumes, pro-perfumes, finishing aids, lime soap dispersants, composition malodor control agents, odor neutralizers, polymeric dye transfer inhibiting agents, crystal growth inhibitors, photobleaches, chelants, anti-tarnishing agents, anti-microbial agents, anti-oxidants, anti-redeposition agents, soil release polymers, electrolytes, pH modifiers, thickeners, abrasives, divalent or trivalent ions, metal ion salts, enzyme stabilizers, corrosion inhibitors, diamines or polyamines and/or their alkoxyates, suds stabilizing polymers, solvents, process aids, fabric softening agents, optical brighteners, hydrotropes, suds or foam suppressors, suds or foam boosters, fabric softeners, anti-static agents, dye fixatives, dye abrasion inhibitors, anti-croaking agents, wrinkle reduction agents, wrinkle resistance agents, soil release polymers, soil repellency agents, sunscreen agents, anti-fade agents, and mixtures thereof.

The term “surfactant” conventionally refers to materials that are surface-active either in the water, the lipophilic fluid,

or the mixture of the two. Some illustrative surfactants include nonionic, cationic and silicone surfactants as used in conventional aqueous detergent systems. Suitable nonionic surfactants include, but are not limited to:

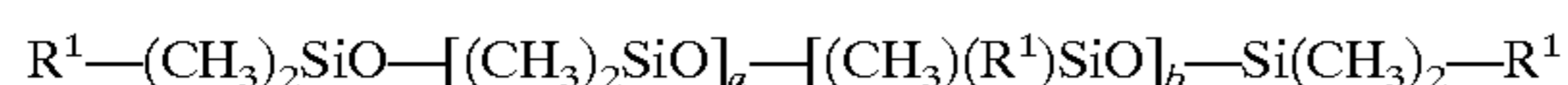
- a) Polyethylene oxide condensates of nonyl phenol and myristyl alcohol, such as in U.S. Pat. No. 4,685,930 Kasprzak; and
- b) fatty alcohol ethoxylates,  $R-(OCH_2CH_2)_aOH$   $a=1$  to 100, typically 12–40,  $R$ =hydrocarbon residue 8 to 20 C atoms, typically linear alkyl. Examples polyoxyethylene lauryl ether, with 4 or 23 oxyethylene groups; polyoxyethylene cetyl ether with 2, 10 or 20 oxyethylene groups; polyoxyethylene stearyl ether, with 2, 10, 20, 21 or 100 oxyethylene groups; polyoxyethylene (2), (10) oleyl ether, with 2 or 10 oxyethylene groups. Commercially available examples include, but are not limited to: ALFONIC, BRIJ, GENAPOL, NEODOL, SURFONIC, TRYCOL. See also U.S. Pat. No. 6,013,683 Hill et al.,.

Suitable cationic surfactants include, but are not limited to dialkyldimethylammonium salts having the formula:

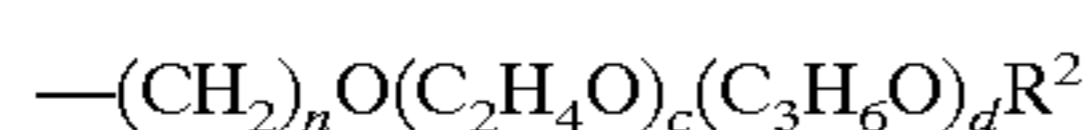


Where each  $R'R''$  is independently selected from the group consisting of 12–30 C atoms or derived from tallow, coconut oil or soy,  $X=Cl$  or  $Br$ , Examples include: didodecyldimethylammonium bromide (DDAB), dihexadecyldimethyl ammonium chloride, dihexadecyldimethyl ammonium bromide, dioctadecyldimethyl ammonium chloride, dieicosyldimethyl ammonium chloride, didocosyldimethyl ammonium chloride, dicoconutdimethyl ammonium chloride, ditallowdimethyl ammonium bromide (DTAB). Commercially available examples include, but are not limited to: ADOGEN, ARQUAD, TOMAH, VARIQUAT. See also U.S. Pat. No. 6,013,683 Hill et al.,.

Suitable silicone surfactants include, but are not limited to the polyalkyleneoxide polysiloxanes having a dimethyl polysiloxane hydrophobic moiety and one or more hydrophilic polyalkylene side chains and have the general formula:



wherein  $a+b$  are from about 1 to about 50, preferably from about 3 to about 30, more preferably from about 10 to about 25, and each  $R^1$  is the same or different and is selected from the group consisting of methyl and a poly(ethyleneoxide/propyleneoxide) copolymer group having the general formula:



with at least one  $R^1$  being a poly(ethyleneoxide/propyleneoxide) copolymer group, and wherein  $n$  is 3 or 4, preferably 3; total  $c$  (for all polyalkyleneoxy side groups) has a value of from 1 to about 100, preferably from about 6 to about 100; total  $d$  is from 0 to about 14, preferably from 0 to about 3; and more preferably  $d$  is 0; total  $c+d$  has a value of from about 5 to about 150, preferably from about 9 to about 100 and each  $R^2$  is the same or different and is selected from the group consisting of hydrogen, an alkyl having 1 to 4 carbon atoms, and an acetyl group, preferably hydrogen and methyl group. Examples of these surfactants may be found in U.S. Pat. No. 5,705,562 Hill and U.S. Pat. No. 5,707,613 Hill, both of which are incorporated herein by reference.

Examples of this type of surfactants are the Silwet® surfactants which are available C K Witco, OSi Division,

Danbury, Conn. Representative Silwet surfactants are as follows.

Name	Average MW	Average a + b	Average total c
L-7608	600	1	9
L-7607	1,000	2	17
L-77	600	1	9
L-7605	6,000	20	99
L-7604	4,000	21	53
L-7600	4,000	11	68
L-7657	5,000	20	76
L-7602	3,000	20	29

The molecular weight of the polyalkyleneoxy group ( $R^1$ ) is less than or equal to about 10,000. Preferably, the molecular weight of the polyalkyleneoxy group is less than or equal to about 8,000, and most preferably ranges from about 300 to about 5,000. Thus, the values of  $c$  and  $d$  can be those numbers which provide molecular weights within these ranges. However, the number of ethyleneoxy units ( $-C_2H_4O$ ) in the polyether chain ( $R^1$ ) must be sufficient to render the polyalkyleneoxide polysiloxane water dispersible or water soluble. If propyleneoxy groups are present in the polyalkyleneoxy chain, they can be distributed randomly in the chain or exist as blocks. Preferred Silwet surfactants are L-7600, L-7602, L-7604, L-7605, L-7657, and mixtures thereof. Besides surface activity, polyalkyleneoxide polysiloxane surfactants can also provide other benefits, such as antistatic benefits, and softness to fabrics.

The preparation of polyalkyleneoxide polysiloxanes is well known in the art. Polyalkyleneoxide polysiloxanes of the present invention can be prepared according to the procedure set forth in U.S. Pat. No. 3,299,112, incorporated herein by reference.

Another suitable silicone surfactant is SF-1488, which is available from GE silicone fluids.

These and other surfactants suitable for use in combination with the lipophilic fluid as adjuncts are well known in the art, being described in more detail in Kirk Othmer's Encyclopedia of Chemical Technology, 3rd Ed., Vol. 22, pp. 360–379, "Surfactants and Detergent Systems", incorporated by reference herein. Further suitable nonionic detergent surfactants are generally disclosed in U.S. Pat. No. 3,929,678, Laughlin et al., issued Dec. 30, 1975, at column 13, line 14 through column 16, line 6, incorporated herein by reference.

The adjunct may also be an antistatic agent. Any suitable well-known antistatic agents used in laundering and dry cleaning art are suitable for use in the methods and compositions of the present invention. Especially suitable as antistatic agents are the subset of fabric softeners which are known to provide antistatic benefits. For example those fabric softeners which have a fatty acyl group which has an iodine value of above 20, such as  $N,N$ -di(tallowoyl-oxyethyl)- $N,N$ -dimethyl ammonium methylsulfate. However, it is to be understood that the term antistatic agent is not to be limited to just this subset of fabric softeners and includes all antistatic agents.

Although the methods and/or compositions utilized in present invention will be described in detail, it should be understood, and one skilled in the art will recognize, that any compositions, processes, and/or apparatuses capable of carrying out the invention could be used.

#### 65 Method

The method of the present invention is directed to attaining improved fabric cleaning in a lipophilic fluid treatment

regimen, and includes the steps of exposing the fabric to a lipophilic fluid and exposing the fabric to a rebuild agent. Optionally but preferably, it may include the step of exposing the fabric to a polar phase.

The polar phase may include water, alcohol, or mixtures thereof. If the polar phase does include water, it preferably comprises at least about 0.5% water by weight of fabric and at most about 10% water by weight of fabric.

The lipophilic fluid may comprise a linear siloxane, a cyclic siloxane, or mixtures thereof. Preferably, the lipophilic fluid is selected from the group consisting essentially of octamethylcyclotetrasiloxane, dodecamethylcyclohexasiloxane, and mixtures thereof. Even more preferably, the lipophilic fluid comprises dodecamethylcyclopentasiloxane. Most preferably, the lipophilic fluid comprises dodecamethylcyclopentasiloxane and is substantially free of octamethylcyclotetrasiloxane. Due to the flash points of the aforementioned siloxanes, the method preferably occurs at less than about 80° C.

While carrying out the method of the present invention, the fabrics may also be exposed to an emulsifier an/or a surfactant either separately or as a result of being contained within the polar phase, the lipophilic fluid, and/or the bleach system. The fabrics may also be exposed to adjunct ingredients selected from the group consisting essentially of enzymes, bleaches, surfactants, fabric softeners, perfumes, antibacterial agents, antistatic agents, brighteners, dye fixatives, dye abrasion inhibitors, anti-crocking agents, wrinkle reduction agents, wrinkle resistance agents, soil release polymers, sunscreen agents, anti-fade agents, builders, chelants, sudsing agents, composition malodor control agents, composition coloring agents, pH buffers, waterproofing agents, soil repellency agents, and mixtures thereof. These adjuncts can also be applied either separately or as a result of being contained within the polar phase, the lipophilic fluid, and/or the rebuild agent.

#### Composition

The composition of the present invention is directed to attaining improved fabric cleaning in a lipophilic fluid treatment regimen, wherein the composition comprises a lipophilic fluid and a rebuild agent. Optionally, the composition can further comprise a polar phase.

If included, the polar phase may include water, alcohol, and mixtures thereof. Also, the polar phase preferably comprises at least about 0.1% water by weight of composition and at most about 5% water by weight of composition.

Further, the polar phase may comprise a buffer to maintain pH.

The composition may contain non-rebuild agents also to stabilize the product during storage prior to delivery in the lipophilic system. Such chelating agents may comprise, but are not limited to, ethylenediaminedisuccinate (EDDS), ethylene diamine tetra acetic acid (EDTA), quaternary ammonia compounds, or 1-Hydroxyethane-1,1-diphosphonic acid (HEDP).

The lipophilic fluid may comprise a linear siloxane, a cyclic siloxane, or mixtures thereof. Preferably, the lipophilic fluid comprises a lipophilic fluid selected from the group consisting essentially of octamethylcyclotetrasiloxane, dodecamethylcyclopentasiloxane, dodecamethylcyclohexasiloxane, and mixtures thereof. More preferably, the lipophilic fluid comprises dodecamethylcyclopentasiloxane. Most preferably, the lipophilic fluid comprises dodecamethylcyclopentasiloxane and is substantially free of octamethylcyclotetrasiloxane.

The bleach system may include oxygen-based bleach, bleach activator and a peroxide source, pre-formed peracid, oxidative bleach enzyme, photo bleach, bleach boosting compounds, metal bleach catalysts, ozone, chlorine dioxide or mixtures of multiple bleach systems. If the bleach system comprises pre-formed peracid the polar phase preferably comprises at least about 1% water by weight of fabric. Preferably, the bleach system has at least about 2 ppm AvO, more preferably at least about 25 ppm AvO, even more preferably at least about 50 ppm AvO, even more preferably at least about 100 ppm AvO. Preferably, the bleach system has at most about 10000 ppm AvO. Most preferably, the bleach system has at least about 100 ppm AvO and at most about 5000 ppm AvO. The bleach system may be within the polar phase and/or within the lipophilic fluid as opposed to being a stand-alone component.

While carrying out the present invention, the fabrics may also be exposed to an emulsifier an/or a surfactant either separately or as a result of being contained within the polar phase, the lipophilic fluid, and/or the bleach system. The fabrics may also be exposed to adjunct ingredients selected from the group consisting essentially of enzymes, bleaches, emulsifiers, surfactants, fabric softeners, perfumes, antibacterial agents, antistatic agents, brighteners, dye fixatives, dye abrasion inhibitors, anti-crocking agents, wrinkle reduction agents, wrinkle resistance agents, soil release polymers, sunscreen agents, anti-fade agents, builders, chelants, sudsing agents, composition malodor control agents, composition coloring agents, pH buffers, waterproofing agents, soil repellency agents, and mixtures thereof. These adjuncts can also be applied either separately or as a result of being contained within the polar phase, the lipophilic fluid, and/or the bleach system.

It will be understood that the methods and/or compositions of the present invention may be combined with other fabric treatments. For example, prior to the application of the lipophilic fluid the fabric articles may be subjected to the particulate removal method described in co-pending application Ser. No. 60/191,965, to Noyes et al., filed Mar. 24, 2000, the relevant parts of which are incorporated herein by reference.

The present invention may be used in a service, such as a dry cleaning service, diaper service, uniform cleaning service, or commercial business, such as a Laundromat, dry cleaner, linen service which is part of a hotel, restaurant, convention center, airport, cruise ship, port facility, casino, or may be used in the home.

The methods and/or compositions of the present invention may be performed in an apparatus that is a modified existing apparatus and is retrofitted in such a manner as to conduct the process of the present invention in addition to related processes.

The methods and/or compositions of the present invention may also be performed in an apparatus, which is not a modified existing apparatus but is one specifically built in such a manner so as to conduct the process of the present invention or may be added to another apparatus as part of a lipophilic fluid processing system. This would include all the associated plumbing, such as connection to a chemical and water supply, and sewerage for waste wash fluids.

Finally, the methods of the present invention may be performed in an apparatus, which is not a modified existing apparatus but is one specifically built in such a manner so as to conduct the process of the present invention and related processes.

An apparatus used to carry out the present invention will typically contain some type of control system. These include



electrical systems, such as, the so-called smart control systems, as well as more traditional electro-mechanical systems. The control systems would enable the user to select the size of the fabric load to be cleaned, the type of soiling, the extent of the soiling, the time for the cleaning cycle. Alternatively, the user could use pre-set cleaning and/or refreshing cycles, or the apparatus could control the length of the cycle, based on any number of ascertainable parameters. This would be especially true for electrical control systems. For example, when the collection rate of lipophilic fluid reaches a steady rate the apparatus could turn its self off after a fixed period of time, or initiate another process for the lipophilic fluid.

In the case of electrical control systems, one option is to make the control device a so-called "smart device". This could mean including, but not limited to, self diagnostic system, load type and cycle selection, linking the machine to the Internet and allowing for the consumer to start the apparatus remotely, be informed when the apparatus has cleaned a fabric article, or for the supplier to remotely diagnose problems if the apparatus should break down. Furthermore, if the apparatus of the present invention is only a part of a cleaning system, the so called "smart system" could be communicating with the other cleaning devices which would be used to complete the remainder of the cleaning process, such as a washing machine, and a dryer.

What is claimed is:

1. A method for treating a fabric article in need of treatment comprising the steps of exposing the fabric article to a rebuild agent-containing composition comprising more than 50% of a lipophilic fluid and a rebuild agent such that the fabric article is treated;

wherein said rebuild agent comprises a polymeric backbone having one or more pendant groups which undergo a chemical change to cause an increase in affinity for fabric.

2. The method according to claim 1 wherein the composition further comprises a polar phase.

3. The method according to claim 2 wherein said polar phase comprises water.

4. The method according to claim 2 wherein said polar phase comprises at least about 0.1% water by weight of fabric.

5. The method according to claim 2 wherein said polar phase comprises at most about 5% water by weight of fabric.

6. The method according to claim 2 wherein said polar phase comprises alcohol.

7. The method according to claim 1 wherein the lipophilic fluid comprises a linear siloxane, a cyclic siloxane and mixtures thereof.

8. The method according to claim 1 wherein said lipophilic fluid comprises a lipophilic fluid selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, dodecamethylcyclohexasiloxane, and mixtures thereof.

9. The method according to claim 8 wherein said lipophilic fluid comprises decamethylcyclopentasiloxane.

10. The method according to claim 8 wherein said lipophilic fluid comprises decamethylcyclopentasiloxane and is substantially free of octamethylcyclotetrasiloxane.

11. The method according to claim 1 comprising the additional step of exposing said fabrics to an emulsifier.

12. The method according to claim 1 comprising the additional step of exposing said fabrics to a surfactant.

13. The method according to claim 1 wherein the method occurs at less than about 80° C.

14. The method according to claim 2 wherein the method occurs at less than about 80° C.

15. The method according to claim 1 wherein said fabric is also exposed to adjunct ingredients selected from the group consisting of enzymes, bleaches, surfactants, fabric softeners, perfumes, antibacterial agents, antistatic agents, brighteners, dye fixatives, dye abrasion inhibitors, anti-crocking agents, wrinkle reduction agents, wrinkle resistance agents, soil release polymers, sunscreen agents, anti-fade agents, builders, chelants, sudsing agents, composition malodor control agents, composition coloring agents, pH buffers, waterproofing agents, soil repellency agents, and mixtures thereof.

16. A fabric treating composition comprising more than 50% of a lipophilic fluid and a rebuild agent comprising a polymeric backbone having one or more pendant groups which undergo a chemical change to cause an increase in affinity for fabric.

17. The composition according to claim 16 wherein said composition further comprises a polar phase.

18. The composition according to claim 17 wherein said polar phase comprises water.

19. The composition according to claim 18 wherein said polar phase comprises at least about 0.1% water by weight of composition.

20. The composition according to claim 18 wherein said polar phase comprises at most about 5% water by weight of composition.

21. The composition according to claim 17 wherein said polar phase comprises alcohol.

22. The composition according to claim 16 wherein said lipophilic fluid comprises a linear siloxane, a cyclic siloxane, or mixtures thereof.

23. The composition according to claim 16 wherein said lipophilic fluid comprises a lipophilic fluid selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, dodecamethylcyclohexasiloxane, and mixtures thereof.

24. The composition according to claim 23 wherein said lipophilic fluid comprises decamethylcyclopentasiloxane.

25. The composition according to claim 23 wherein said lipophilic fluid comprises decamethylcyclopentasiloxane and is substantially free of octamethylcyclotetrasiloxane.

26. The composition according to claim 16 further comprising adjunct ingredients selected from the group consisting of enzymes, bleaches, emulsifiers, surfactants, fabric softeners, perfumes, antibacterial agents, antistatic agents, brighteners, dye fixatives, dye abrasion inhibitors, anti-crocking agents, wrinkle reduction agents, wrinkle resistance agents, soil release polymers, sunscreen agents, anti-fade agents, builders, non-rebuild agents, sudsing agents, composition malodor control agents, composition coloring agents, pH buffers, waterproofing agents, soil repellency agents, and mixtures thereof.